

Durability of Intercalated Graphite Epoxy Composites in Low Earth Orbit

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INTRODUCTION

One of the important trends in the manufacture of aerospace structures has been towards replacing conventional metallic components with high performance polymer matrix composites. When high strength and stiffness are required carbon fiber polymer matrix composites have often been the material of choice. Mass savings in excess of 20 percent are common, resulting in significant performance improvements.

There has been resistance, however, to substituting composites in structural components which also have an electrical role, such as electromagnetic interference (EMI) shielding enclosures and ground planes, because the electrical resistivity of carbon fiber polymer composites is typically three orders of magnitude higher than that of metals. High performance pitch-based graphite fibers, such as Thornel P-100 (Amoco) have lower resistivity, and so reduce the margin to two orders of magnitude. The process of intercalation, the insertion of guest atoms or molecules between the graphene planes within the fiber, can reduce the resistivity by another factor of five (ref. 1).

Studies have shown that applications such as EMI shielding may indeed be feasible when intercalated pitch-based fiber composites are used¹. Such composites could reduce the mass of satellite power systems by as much as 17 percent and provide similar mass savings within the communications systems. The combined mass of the power and communications systems typically account for 40 percent of the spacecraft mass. Reducing the mass of these systems would provide the opportunity to significantly increase payload capacities (ref. 2)

One of the reservations about using intercalated graphite fiber composites has been the possibility of chemical instability of the fibers. Since the most stable intercalates are strong oxidizing agents (such as bromine) the concern is justified. Bromine vapor is corrosive enough that even at low concentrations it could wreak havoc on an electrical system. Although studies have shown that bromine intercalated pitch-based fibers are stable under ambient conditions, high humidity, high vacuum, and temperatures from 150 to 300 °C (depending on the fiber host) for several years², intercalated graphite fiber composites had not been directly exposed to the launch and space environments.

The low Earth orbit (LEO) environment experienced by spacecraft is particularly hostile for organic compounds, such as intercalated graphite composites. There is rapid thermal cycling as the spacecraft flies in and out of the Earth's shadow. Short wavelength ultraviolet light which is blocked from the Earth's surface by the atmosphere (vacuum ultraviolet) is not blocked, and facilitates many chemical reactions of organic compounds. Perhaps most significantly at altitudes between 300 to 500 km, where Space Shuttles operate, the most common chemical specie is atomic oxygen (ref. 3). This atomic oxygen is the outer remnant of the Earth's atmosphere, and as such follows the 24 hr rotational motion of the Earth. Shuttle however, orbits about the Earth about once every 90 min (1 000 m/hr), resulting in significant relative motion between the spacecraft and the atomic oxygen. This leads to an average relative kinetic energy of 4 to 5 eV for the atomic oxygen. This highly energetic, reactive oxygen quickly attacks any compound that is susceptible to reaction with oxygen.

The consequence of this environment is that any surface which is susceptible to atomic oxygen attack must be protected. The strategy which has proved most successful is to apply thin films of materials which are already highly

oxidized. Sputter deposited SiO_2 and Al_2O_3 films which are a few tenths of a micrometer thick have proved effective for protecting smooth surfaces.

The effects of the LEO environment on materials have been subject to specific investigation by NASA for more than a decade. An important component has been the Evaluation of Oxygen Interaction with Materials (EOIM) experiments flown on the space shuttle (ref. 3). The first (EOIM-1) was flown in November, 1982 on the STS-5 mission. Forty-five material samples were exposed for 44 hr to ram atomic oxygen at an altitude of 300 km. The resulting atomic oxygen fluence was determined to be 1×10^{20} atoms/cm². EOIM-2 was flown aboard STS-8 in August 1983. Over 300 samples were flown for 42 hours at 250 km, resulting in an atomic oxygen fluence of 3.5×10^{20} atoms/cm². The most recent of the Evaluation of Oxygen Interaction with Materials experiment (EOIM-3) was launched on the Space Shuttle Discovery (STS-46) on July 31, 1992. On August 6, at 5:30 AM EST the experiment was initiated where the materials were exposed to atomic oxygen in the ram direction for 42 hr and an altitude of 230 km. The resulting fluence was 2.6×10^{20} atoms/cm². EOIM-3 carried many active and passive materials experiments including more than 300 samples, including six that were designed to evaluate the durability of inter-calated graphite composites to LEO. Additionally, control samples were subjected to simulated LEO conditions in an attempt to determine how closely ground based testing simulates the true LEO environment for these materials.

EXPERIMENTAL

Three grades of Amoco pitch-based graphite fibers, Thornel P-100, P-75, and P-55 were selected for this study because of their availability and the large data base available on the properties of their intercalation compounds. Bromine was selected as the intercalate because it forms reproducible and very stable intercalation compounds with all three types of fibers. Mutual Industries (Philadelphia, PA) wove P-55 and P-75 fibers in a five harness satin weave with 11×11 , 6000 filament tows per inch. Fabric Development, Inc. (Quakertown, PA) wove P-100 fabric in a 10×10 , 2000 filament tows per inch. A plain weave was used to minimize fiber breakage during weaving. Four ply 0 to 90° laminar composites were fabricated by Rohr Industries (Chula Vista, CA) using Hysol-Grafil EAG101-1 film epoxy. The details of the intercalation and composite fabrication and characterization have been reported elsewhere (ref. 3).

Silicon dioxide (SiO_2) coatings were applied to some of the samples by ion beam sputtering using an Ion Tech dual ion source with argon ions and an SiO_2 target. Circular disks (2.50 cm) were cut from the composite sheets using electron discharge machining (EDM). These fit into the sample holder in a hexagonal array as shown in fig. 1. These were flown in tray 6 as samples 6-40 to 6-45.

Properties of the six flight samples are summarized in Table I. In addition to the flight samples there was a complete set of back-up samples, which traveled with the flight samples, but were not mounted. There were also two sets of control samples which were prepared at the same time, but which stayed at the lab during the flight.

TABLE I.—Samples flown on STS-46

Sample	Fiber type	Intercalate	Coating	Br ₂ trap
6-40	P-100	none	none	none
6-41	P-100	Br ₂	none	none
6-42	P-100	Br ₂	SiO ₂	none
6-43	P-100	Br ₂	none/SiO ₂	Cu
6-44	P-75	Br ₂	none/SiO ₂	Cu
6-45	P-55	Br ₂	none/SiO ₂	Cu

Sample 6-40 was a pristine P-100 composite with no coating. To our knowledge P-100 composites had not been flown before, but it was expected to behave similarly to other carbon fiber epoxy composites that have been flown in the past. This sample was to act as a baseline to which the intercalated fiber samples could be compared.

Sample 6-41 was a bromine intercalated P-100 composite sample. Its comparison with sample 6-40 would show the effects of intercalation on the durability of an unprotected sample in LEO. It would also show whether subjecting an intercalated composite sample to the LEO environment would deintercalate the sample or change its physical or chemical properties.

Sample 6-42 was a bromine intercalated P-100 composite sample that was protected with 0.10 μm of SiO_2 . Its comparison with sample 6-41 would indicate whether intercalated graphite composites could be protected using the same techniques used for other carbon fiber polymer composites.

Samples 6-43, 6-44, and 6-45 were similar except that the composites were made with intercalated P-100, P-75, and P-55 fibers respectively. One half of each of these samples was coated with SiO_2 , so that the effect of the coating could be observed directly. In addition, an expanded copper foil screen was placed over each of these samples. This was done to provide a "bromine trap." If bromine was released from the composites the bromine would be expected to react with the copper and could be detected in the post-flight analysis. A photo of one of these samples is shown in figure 2.

Optical images with a Kodak MP-4 camera were obtained before and after the flight. Masses of the samples (both pre-flight and post-flight) were measured using a Mettler H315 analytical balance. Reflectance over the 250 to 2500 \AA wavelength range was determined using a Perkin-Elmer Lambda-9 UV-Vis spectrophotometer with an integrating sphere. Electrical conductivity was determined using a Leighton Electronics 1010A eddy current conductivity device which was factory modified to operate at 55.55 kHz, and calibrated with NIST standards. Analysis using a scanning electron microscope (SEM) was carried out using a Cambridge 200 which allowed the sample to be imaged either in transmission or backscatter modes. This instrument was also fitted with energy dispersive spectroscopy (EDS) which enabled elemental analysis at the resolution of the microscope. As much of the SEM analysis as possible was done on the samples as received, but the high resolution work required about 20 nm of gold to be sputtered onto the surfaces.

Approximate values for the erosion yields (mass loss per impinging oxygen atom) were also determined using the SEM. Dust particles adhered to the surface of the composite protect small portions of the composite from erosion, and the depth of the material eroded is directly measured from the micrographs. In addition to the dust particles, the interfaces where the sample was protected by the sample holder, the SiO_2 coatings, or the bromine traps, could also be utilized. The technique is fraught with assumptions, but given enough data points, consistent values for the erosion depths do emerge.

Control samples were subjected to ground based atomic oxygen simulation in a Plasma Prep 2 radio frequency plasma asher. These samples received an oxygen fluence equal to that received by the flight samples as determined by Kapton witness coupons (ref. 6).

RESULTS AND DISCUSSION

Images of the samples before and after the flight test are shown in figure 3. The outer ring on each of the samples is where the surface was protected by contact with the sample holder, and appears to be very similar to the pre-flight condition. It is immediately apparent that the unprotected portions of the samples were changed by the LEO environment. Those areas have a velvet texture and appear to be darker than the unexposed areas. This same phenomenon was reported by Slomp, et al. for graphite epoxy composites flown on STS-8 in EOIM-2 (ref. 4). There are no obvious differences either between the pristine and intercalated samples (6-40 and 6-41), or among the different grades of pitch-based fiber (samples 6-43, 6-44, and 6-45). Those portions of the sample which were under the SiO_2 coating are visually indistinguishable from those areas protected by the sample holder (note especially the fully protected sample 6-42). The cross-hatch pattern visible in samples 6-43, 6-44, and 6-45 corresponds to the copper screen which was placed over each of those samples as a bromine trap.

These qualitative observations were quantified using the reflectance measurements. Figure 4 shows the total hemispherical reflectance from 250 to 2500 nm before and after LEO exposure for the pristine P-100 composite (6-40), the bromine intercalated composite (6-41) and the protected bromine intercalated composite (6-42). A change in total reflectance factor, ΔR was calculated point by point for each of these samples using:

$$\Delta R = \frac{R_{\text{after}} - R_{\text{before}}}{R_{\text{before}}}$$

The values calculated are shown in table IV.

TABLE IV.—CHANGE IN TOTAL
HEMISPHERICAL REFLECTANCE
ON EXPOSURE TO LEO

Sample	ΔR
6-40	-0.64 ± 0.03
6-41	-0.51 ± 0.09
6-42	0.17 ± 0.12

There was a wavelength dependence in the reflectance change for the pristine samples with the low frequency ΔR being about -0.4 and in the high frequency about -1.0. The ΔR of the intercalated fiber composite showed no frequency dependence except for the 250 to 500 nm region. The ΔR of the SiO_2 protected composite showed a slight increase in the reflectance which increases from negligible (between 900 and 1000 nm) to (0.04 at 2500 nm). The important technological conclusion however, is that all three composites maintained low reflectance values (below 0.2) over the entire measured spectrum. These results are consistent with other unprotected and protected carbon fiber-polymer composites which have been exposed to the LEO environment³.

The mass of each of the samples was measured pre-flight and post-flight and compared. Although there were indications from the appearance and the optical reflectance data that there were mass losses, Table V shows that they were below our detection limit. Composite samples readily absorb water from the atmosphere, and the uncertainty of the mass due to absorbed water was larger than that lost due to space exposure. The mass losses were estimated from the erosion apparent in the SEM analysis (discussed below), but the uncertainty of knowing the original surface morphology on a microscale introduces large errors into any calculation. That the mass losses were very small is the only statement that can be made with confidence.

TABLE V.—MASS OF SAMPLES FLOWN OF STS-46

Sample	m_{initial} grams	m_{final} grams	$m_{\text{final}}/m_{\text{initial}}$
6-40	0.5842	0.5850	1.001
6-41	0.7405	0.7401	0.9995
6-42	0.7452	0.7482	1.004
6-43	0.7497	0.7515	1.002
6-44	0.9050	0.9058	1.001
6-45	0.8886	0.8928	1.005

The resistivity of the samples before and after the flight is shown in Table VI. There were no significant changes, with the possible exception of sample 6-40. This is the pristine P-100 composite, and no plausible explanation for its increase has been reached. These are critical measurements because the principal motivation for using intercalated graphite fiber composites is their low resistivity. The fact that the intercalated fiber composites are unchanged is also a good indication that large amounts of intercalate were not lost, since a small amount of intercalate loss increases the resistivity of the fibers dramatically.

The pristine P-100 composite sample, 6-40, when imaged with the SEM showed evidence of being etched by the atomic oxygen on orbit (fig. 5). The ram direction is apparent by the direction of the cone-shaped structures. This type of structure has been seen before in graphite-epoxy composites subjected to LEO conditions (ref. 10).

When the pristine flight sample, 6-40, (fig. 5) is compared to the intercalated sample, 6-42, (fig. 6), it is apparent that bromine intercalation has no major affect of the degradation of the composite in LEO. The sizes and shapes of the etch artifacts are qualitatively the same. Further, when the unprotected sides of samples 6-43, 6-44, and 6-45, are compared (fig. 7), there are no major differences among the degradation of composites made from the three grades of graphite fibers. To a first approximation, neither fiber type nor intercalation affect the degradation of graphite-epoxy composites in LEO.

TABLE VI.—RESISTIVITY OF SAMPLES FLOWN ON STS-46

Sample	ρ_{initial} $\mu\Omega\text{-cm}$	ρ_{final} $\mu\Omega\text{-cm}$	$\rho_{\text{final}}/\rho_{\text{initial}}$
6-40	1420	1695	1.194
6-41	352	350	0.994
6-42	362	369	1.019
6-43	362	363	1.003
6-44	910	945	1.038
6-45	1520	1529	1.006

When the SiO_2 protected intercalated sample, 6-42, (fig. 8) is compared to the unprotected intercalated sample (fig. 6) the effectiveness of the coating can be judged. The sample looked essentially the same as the unexposed samples. Further, comparing the protected sides of 6-43, 6-44, and 6-45, (fig. 9) it can be seen that changing fiber types does not alter the coating performance.

Erosion yields for the samples are shown in table VII. Sample 6-42 and the sides of samples 6-43, 6-44, and 6-45 which were protected by the SiO_2 coating showed no noticeable erosion. The epoxy matrix showed an average of about $3.5 \times 10^{-24} \text{ cm}^3$ of matrix lost for every atomic oxygen impact. The graphite fibers were somewhat more durable, with an average erosion yield of about one-fourth ($0.85 \times 10^{-24} \text{ cm}^3/\text{atom}$) that of the matrix. As a result, the matrix tends to erode away and leave the fibers on the surface connected to the composite only by the matrix that is shadowed by the fiber. Both the fiber and the matrix tend to erode in such a way as to form cone-shaped structures, but the appearance of matrix structures is more complex, with holes and curled points. Intercalation did not appreciably effect the erosion yield of the fiber. Though it is interesting to note that the highest erosion yield was for the pristine fiber, within the error the yields were the same. The erosion yield calculated under much more controlled conditions for pyrolytic graphite was $0.65 \times 10^{-24} \text{ cm}^3/\text{atom}$, and EOIM-1 found the erosion yield of pyrolytic graphite to be $1.2 \times 10^{-24} \text{ cm}^3/\text{atom}$.⁶

Given the erosion yields above, the total mass loss (Δm) for an unprotected sample can be from

$$\Delta m = (fYFAd)_{\text{fiber}} + (fYFAd)_{\text{matrix}}$$

where f is the volume fraction (0.48 and 0.52 for fiber and matrix respectively), Y the erosion yield, F the fluence of atomic oxygen, A the sample area exposed (4.9 cm^2) and d is the mass density (2.293 and 1.265 for fiber and matrix respectively). This equation assumes that the eroded layer composition is the same as the bulk, which is probably a poor assumption. The surface is known to be resin rich, with at least a thin ($1 \mu\text{m}$) layer of matrix resin covering all of the surface. Given the uncertainties in the rest of the factors, this effect was considered to be below the error. The total mass lost from an unprotected sample was calculated to be about 1.2 mg. This is within the error introduced by the fluctuation of the water content within the composites. The mass loss gives an average etch depth of $4.8 \mu\text{m}$.

Samples exposed to the plasma asher induced simulated LEO environment show similar amounts of erosion, but the morphology of the degradation is different (fig. 10). Whereas the flight samples show a cone-shaped degradative morphology, in the asher a more rounded, pillow-shaped morphology appears. This has been seen in samples before and has been attributed to the flight geometry providing oxygen from an essentially unidirectional source (ram direction through the atmosphere), where as the asher is omni-directional (ref. 9).

One of the most important aspects of this series of experiments was to determine whether bromine would outgas from the intercalated fibers and corrode electrical components. Bromine is not found in detectable quantities on the surface of the fibers, but in a gradient that is at a maximum in the center of the fibers (ref. 11). However, even within the 40 hr period of LEO exposure, some individual fibers were eroded nearly half-way through, so some bromine must have been lost to the environment around the spacecraft. No detectable amount of bromine was found on any of the three copper traps using EDS. A small concentration of oxygen was the only impurity found on the traps.

TABLE VII.—EROSION YIELDS FOR SAMPLES FLOWN ON STS-46

	# of matrix photos	AVG erosion yield matrix (cm ³ /atom)	# of fiber photos	AVG erosion yield fiber (cm ³ /atom)
6-40 Pristine P-100	6	$3.5 \times 10^{-24} \pm 0.6$	11	$1.0 \times 10^{-24} \pm 0.4$
6-41 P-100+Br	7	$4.1 \times 10^{-24} \pm 0.9$	11	$0.8 \times 10^{-24} \pm 0.4$
6-42 P-100+Br+SiO ₂	0	none	0	none
6-43 P-100+Br ¹	---	-----	---	-----
6-44 P-75+Br ²	7	$3.9 \times 10^{-24} \pm 0.5$	11	$0.7 \times 10^{-24} \pm 0.2$
6-45 P-55+Br ²	7	$2.8 \times 10^{-24} \pm 0.5$	11	$0.9 \times 10^{-24} \pm 0.4$

¹Erosion expected to be same as 6-41.²Erosion yield for unprotected side, protected side showed no erosion.

EDS and backscattering were used to determine if bromine leached from the fibers into the epoxy. This scenario was thought unlikely since there is no bromine on the fiber surface, but since the erosion yield of the epoxy was four times that of the fibers, it has the potential to be a potent mechanism for releasing bromine during erosion. The EDS and backscattering results are shown in fig. 11, and demonstrate conclusively that the bromine is confined to the fiber.

This raises the question of the fate of bromine that was once present in the fibers which were eroded. Since it is expected that free bromine would have reacted with the copper traps, the bromine must be released in a chemically inactive form, perhaps as brominated carbon oxides (Br₂CO, etc.), though there are no data to support this speculation.

CONCLUSIONS

Bromine intercalated graphite fiber epoxy composites were fabricated and flown on the Space Shuttle Discovery as part of the EOIM-3 experiment on STS-46. They were subjected to 42 hr of ram atomic oxygen which corresponded to a fluence of about 2.6×10^{20} atoms/cm². Unprotected samples degraded with a matrix erosion yield of about 3.5×10^{-24} cm³/atom, and a fiber erosion yield of about 0.85×10^{-24} cm³/atom. The bromine intercalated erosion was indistinguishable from the pristine composite erosion. There was also no erosion yield differences among the various grades of intercalated graphite fibers. The mass loss for all of the samples was below the detectable limit using a conventional analytical balance, but was calculated from the erosion yield to be about 1.2 mg. Total reflectance of these samples decreased by a factor of 50 to 65 percent. The electrical conductivity of the samples was unchanged. Bromine was found to remain within the fibers with no detectable amounts leaching into the epoxy or out onto copper bromine traps. SiO₂ coatings were found to be effective in preventing the erosion of the composites without adversely affecting conductivity or reflectance. Control samples exposed to the same fluence in a plasma asher were also found to exhibit no detectable changes in mass loss and conductivity, but the surface morphology was quite different than that built up during low Earth orbit.

This experiment allays fears that intercalated graphite fiber composites might outgas large quantities of corrosive materials which would destroy sensitive electronics in the vicinity of the composites. It also shows that there is no particular oxidation resistance conferred on composites by intercalation. Rather, it reinforces the perception that the only significant difference between pristine and intercalated graphite fiber composites is the latter's enhanced electrical conductivity.

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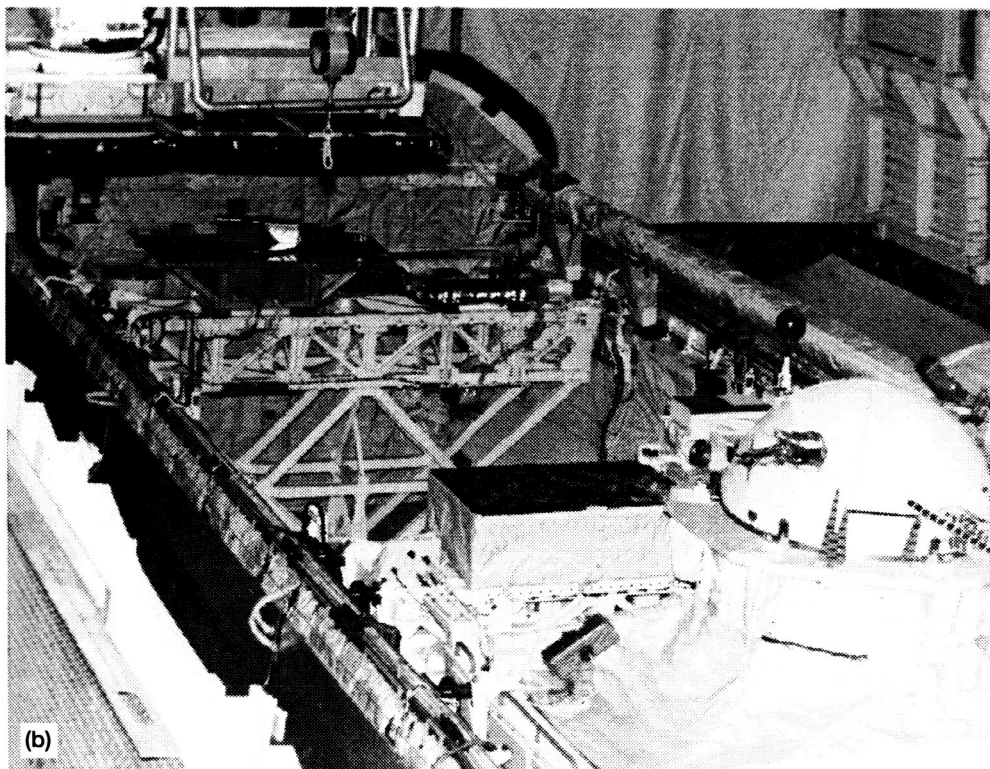
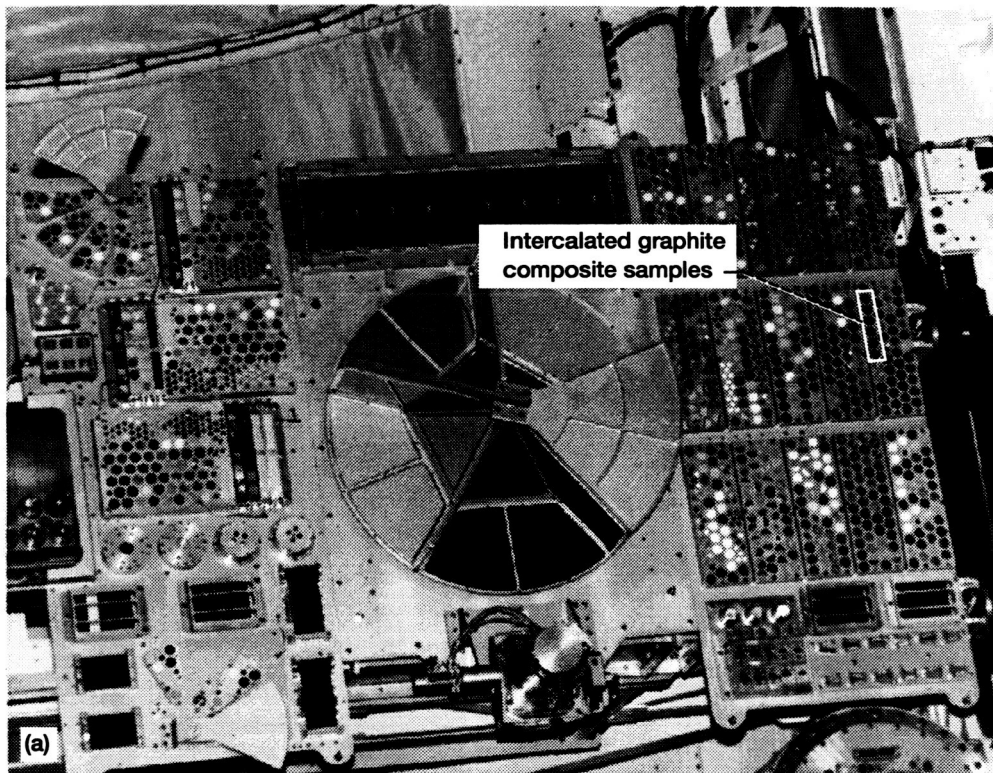


Figure 1.—EOIM III sample trays with intercalated graphite composite samples (a) indicated; (b) in shuttle bay.

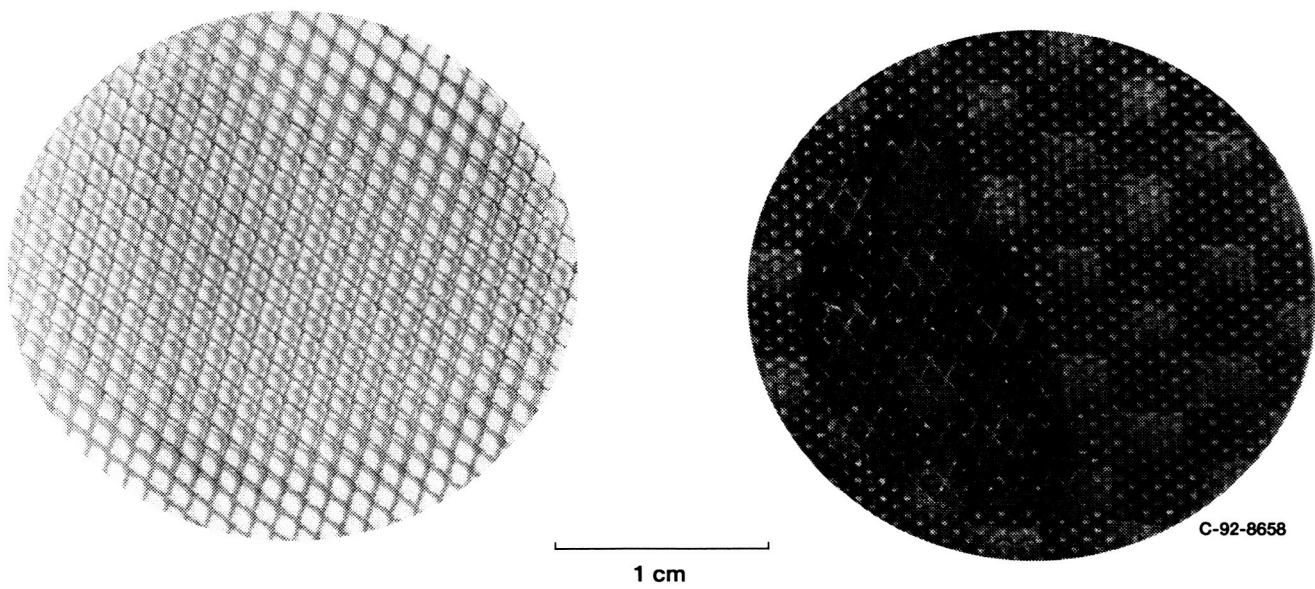


Figure 2.—Flight sample 6-43 showing the configuration of the protective film and the bromine trap.

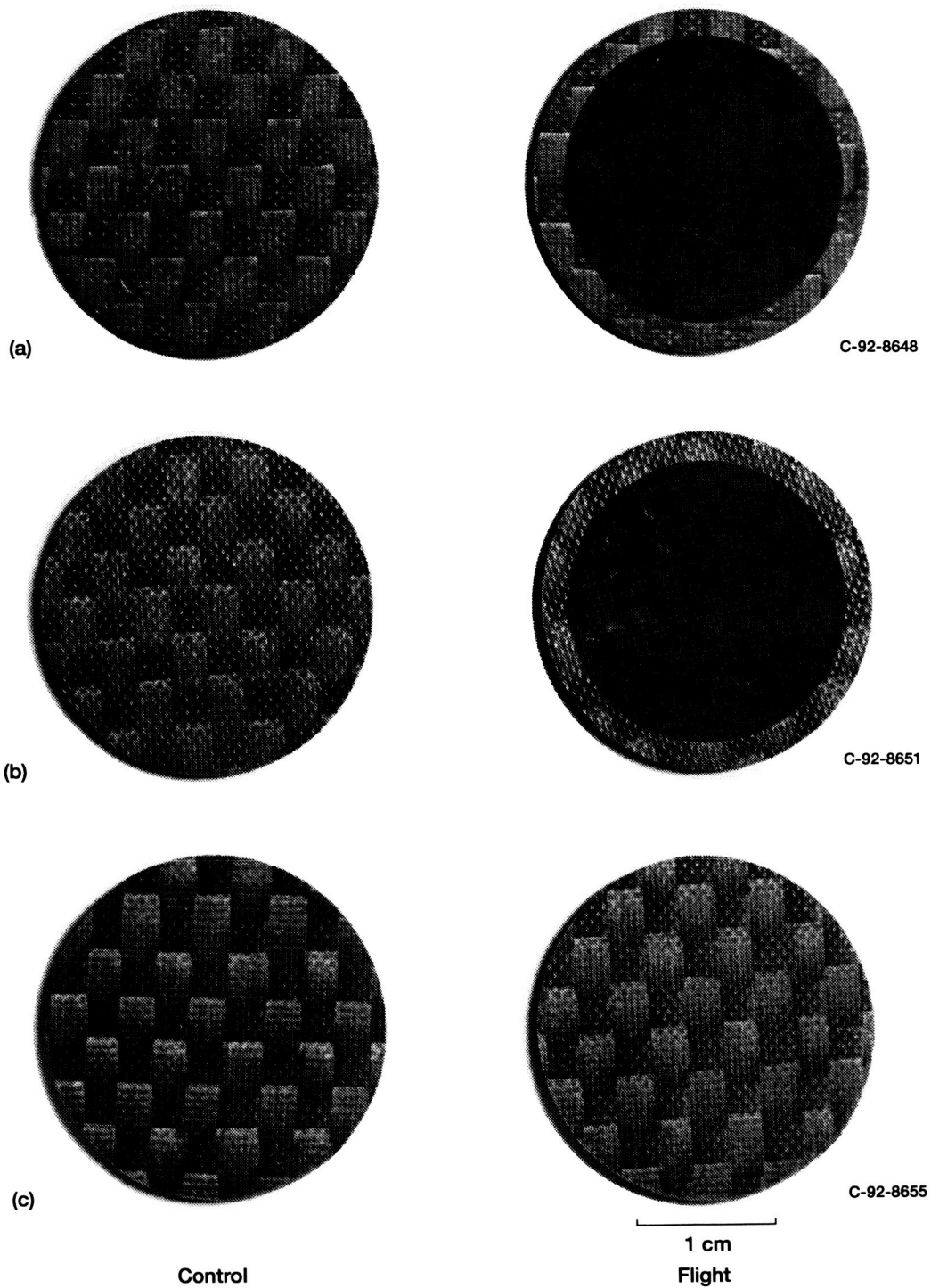


Figure 3.—Optical images of the six flight samples after exposure to LEO and their control samples. (a) Pristine P-100. (b) P-100 + Br. (c) P-100 + Br (coated w/ SiO_2).

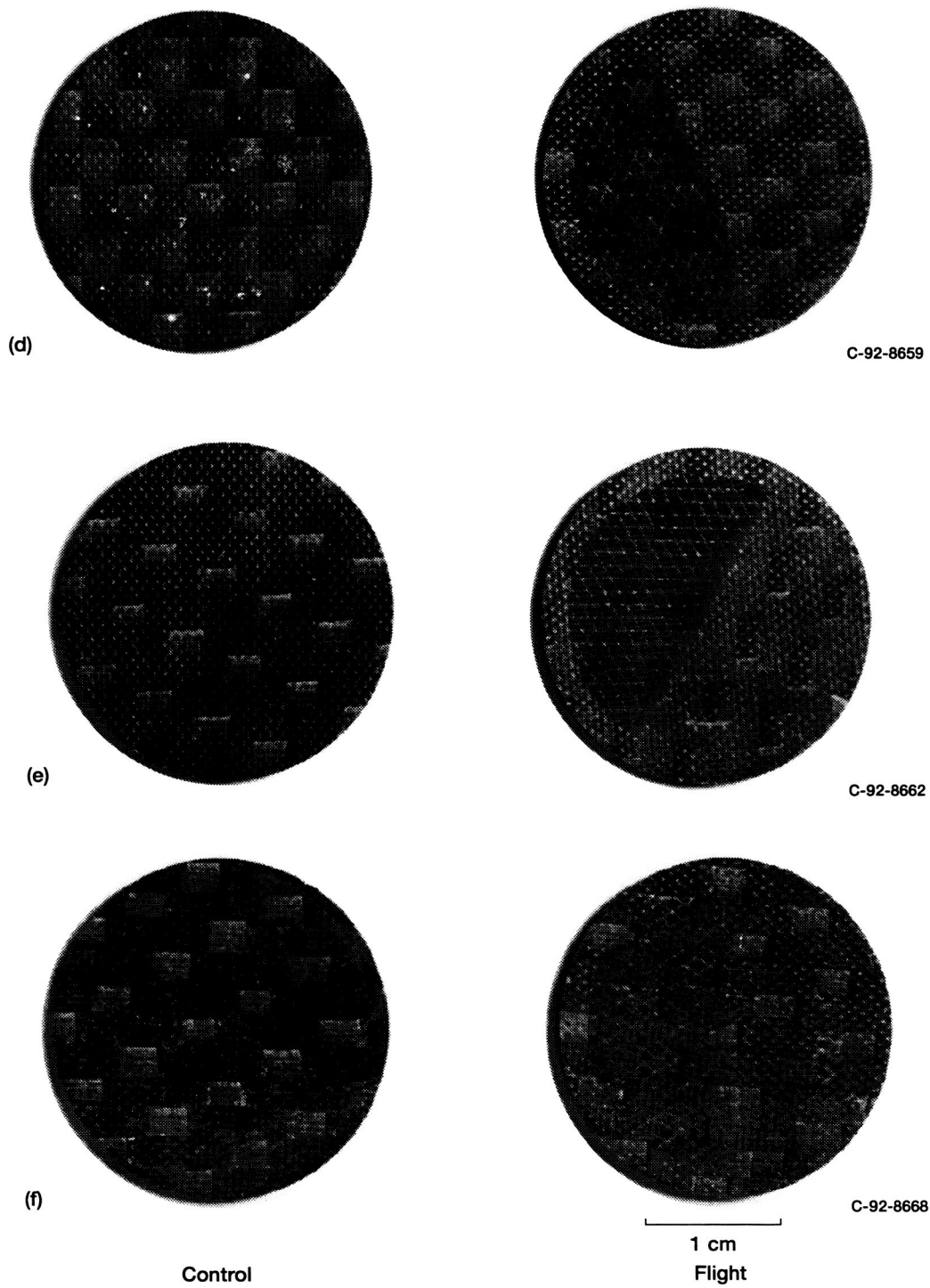


Figure 3.—Continued. (d) P-100 + Br (1/2 coated w/SiO₂). (e) P-75 + Br(1/2 coated w/SiO₂).
(f) P-55 + Br (1/2 coated w/ SiO₂).

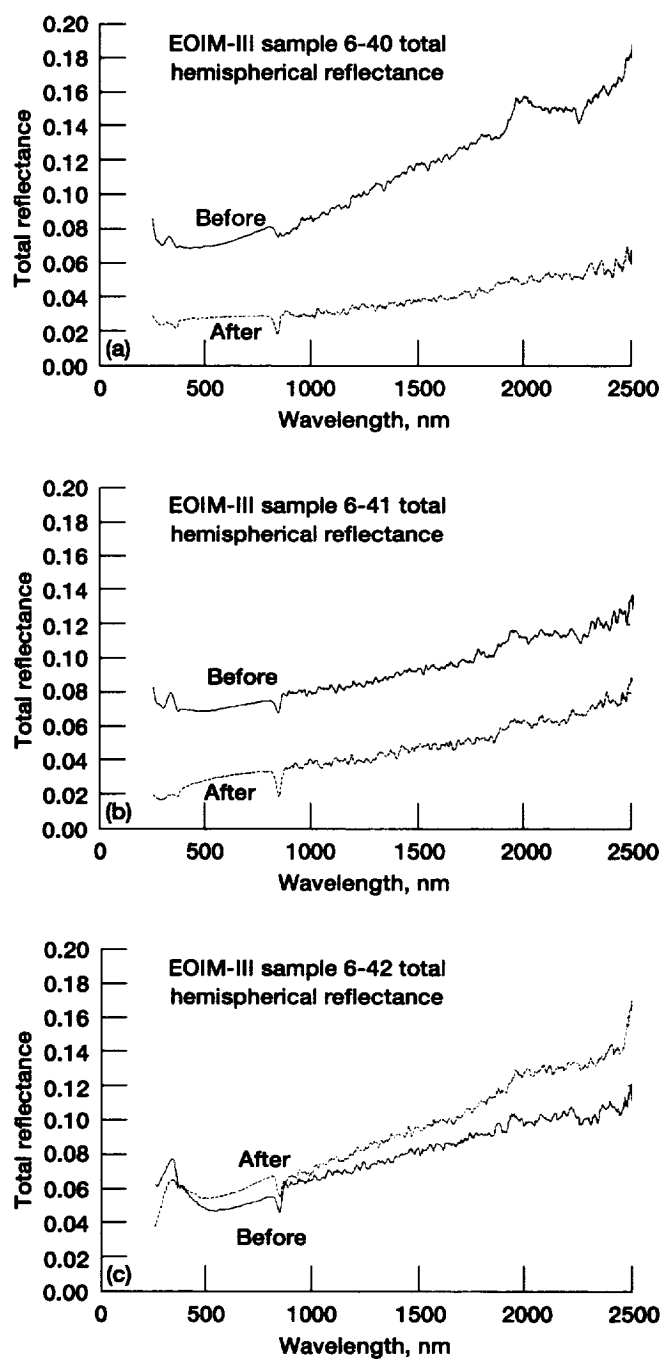


Figure 4.—Reflectance of; (a) pristine P-100 composites, (b) bromine intercalated P-100 composites, (c) SiO₂ protected bromine intercalated P-100 composites; before and after LEO exposure.

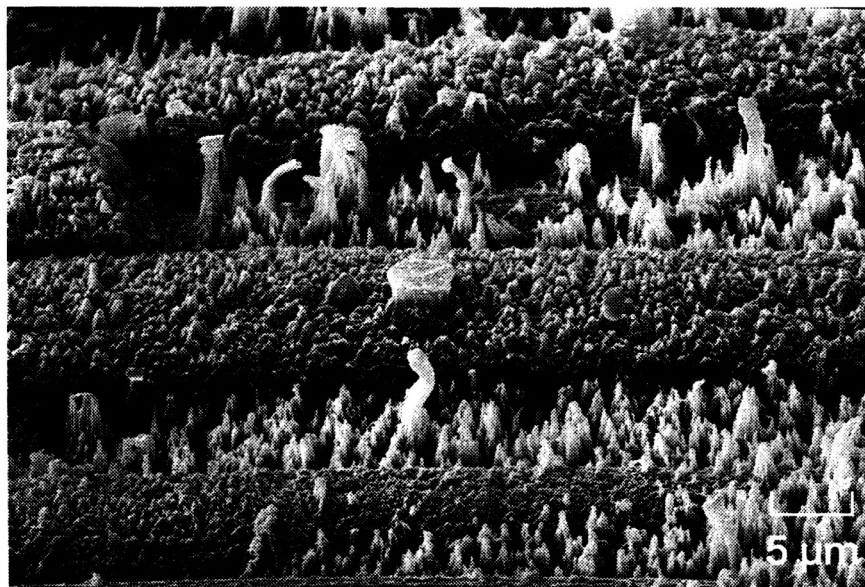


Figure 5.—Photomicrograph of pristine P-100 composite after LEO exposure.

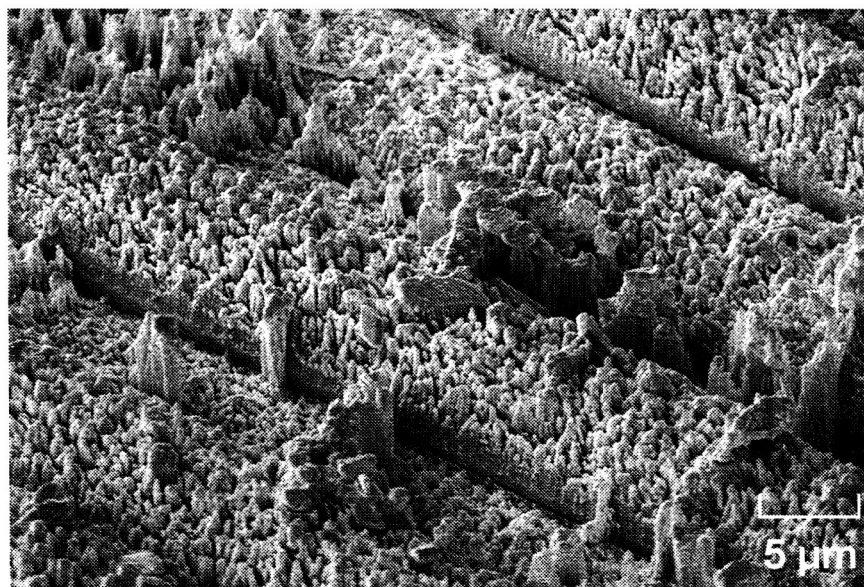


Figure 6.—Photomicrograph of bromine intercalated P-100 composite after LEO exposure.

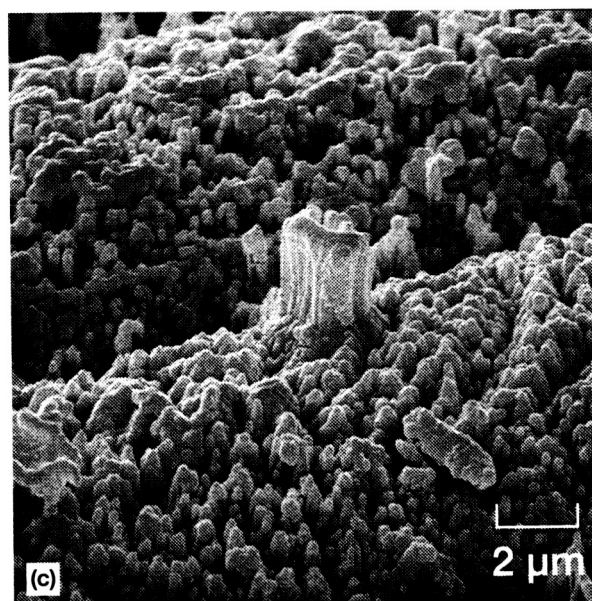
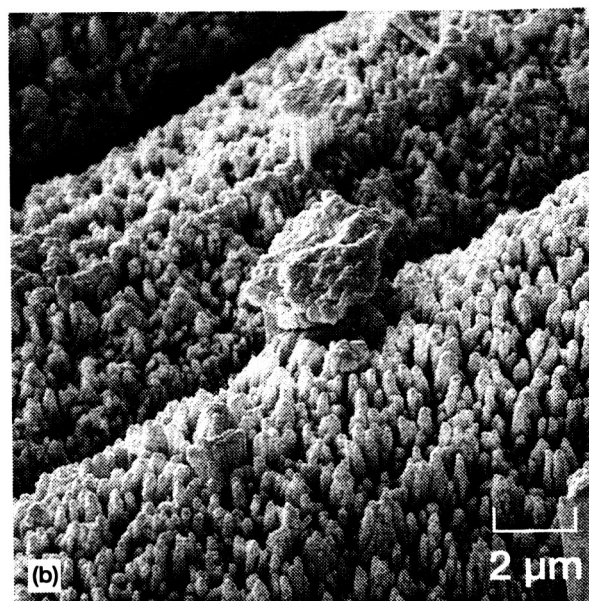
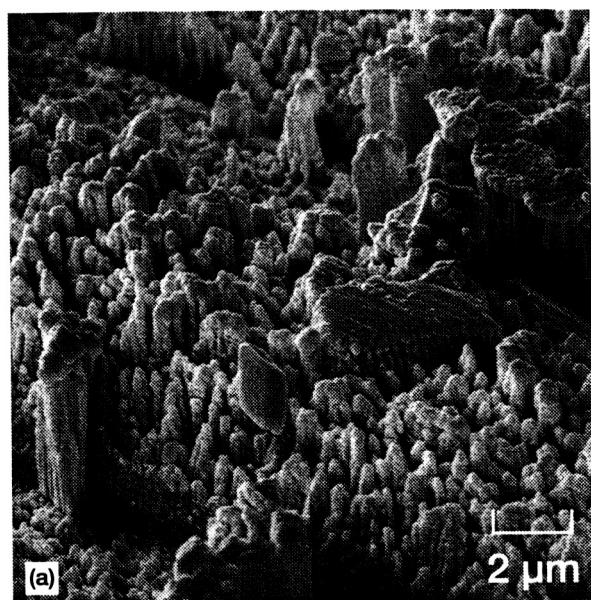


Figure 7.—Photomicrograph of unprotected bromine intercalated (a) P-100; (b) P-75; (c) P-55; composite after LEO exposure.

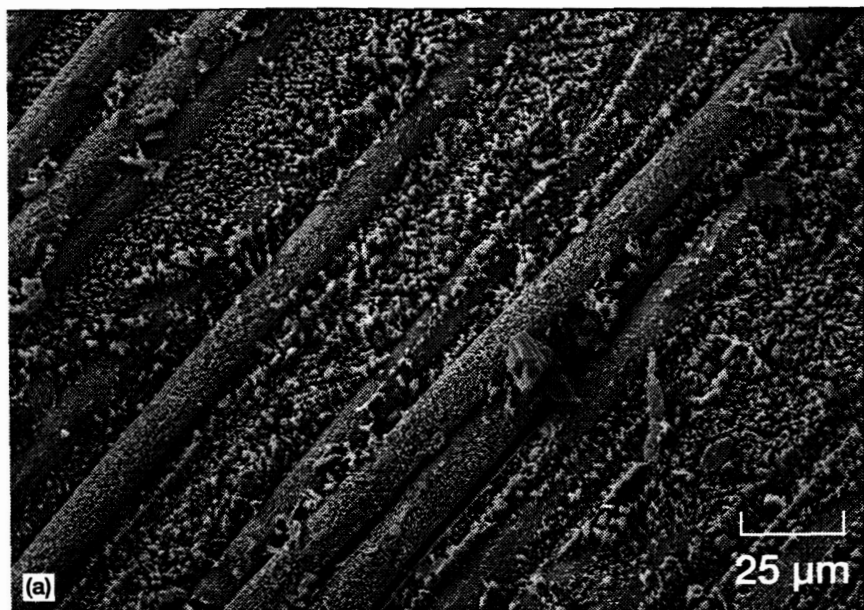


Figure 8.—Photomicrograph of (a) P-100 + Br (b) P-100 + Br (coated w/SiO₂) composite after LEO exposure.

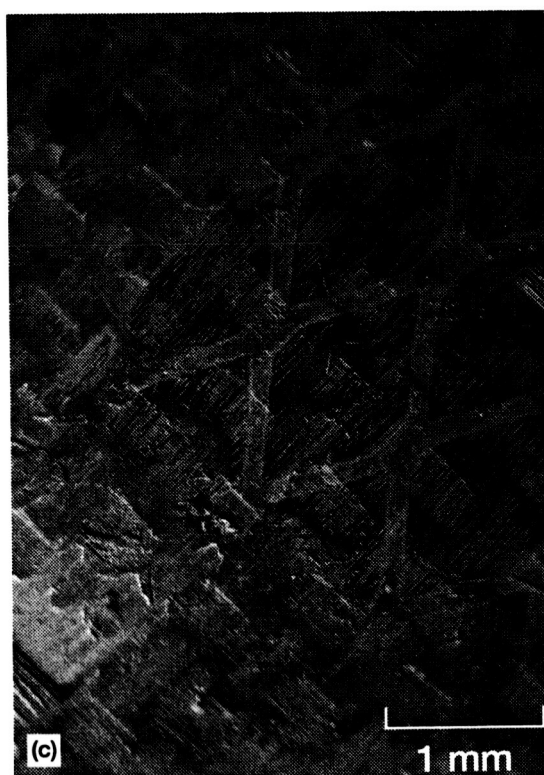
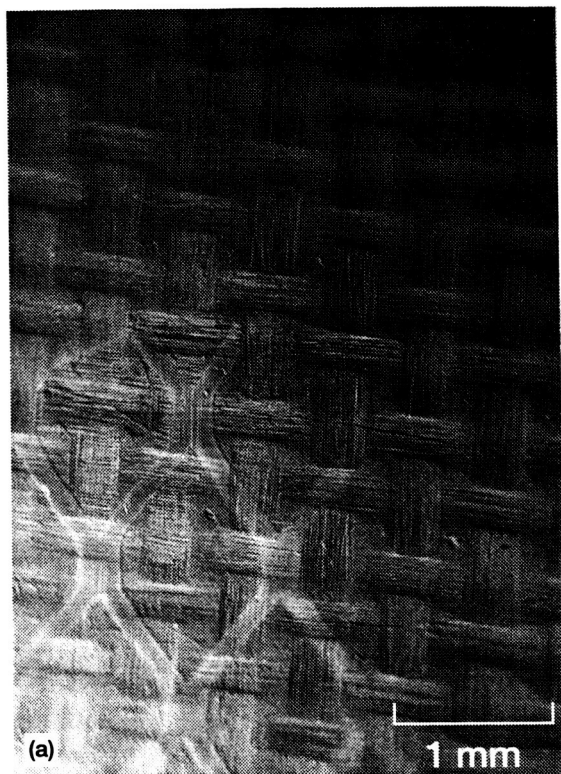


Figure 9.—Photomicrograph of SiO_2 protected bromine intercalated (a) P-100; (b) P-75; (c) P-55; composite after LEO exposure.

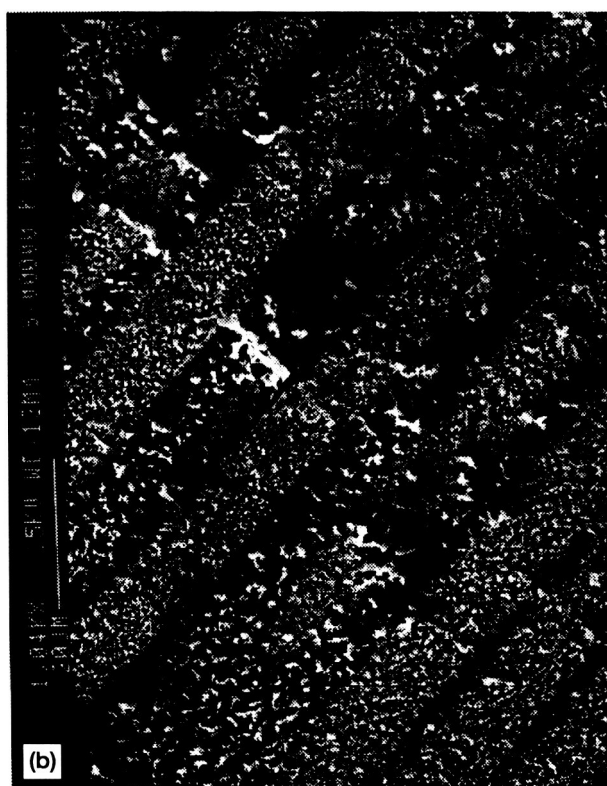
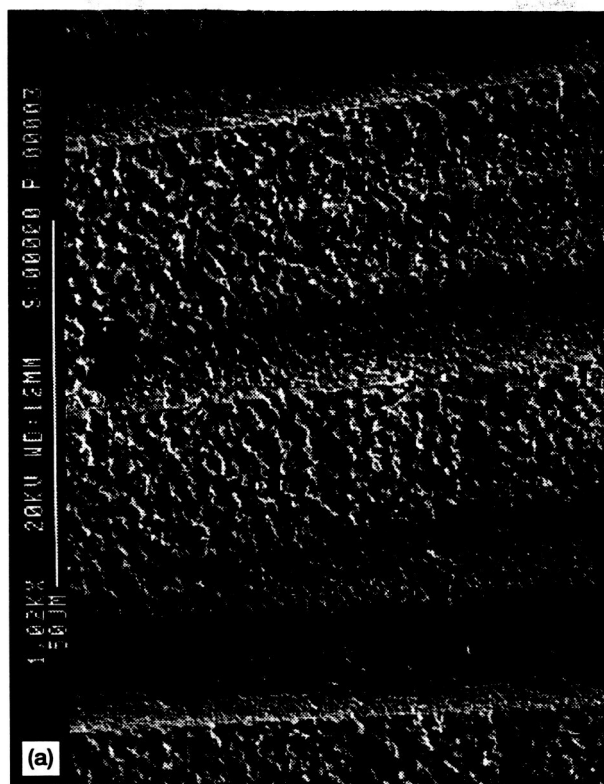


Figure 10.—Photomicrograph of bromine intercalated P-100 composite (a) after LEO exposure; (b) after plasma asher exposure.

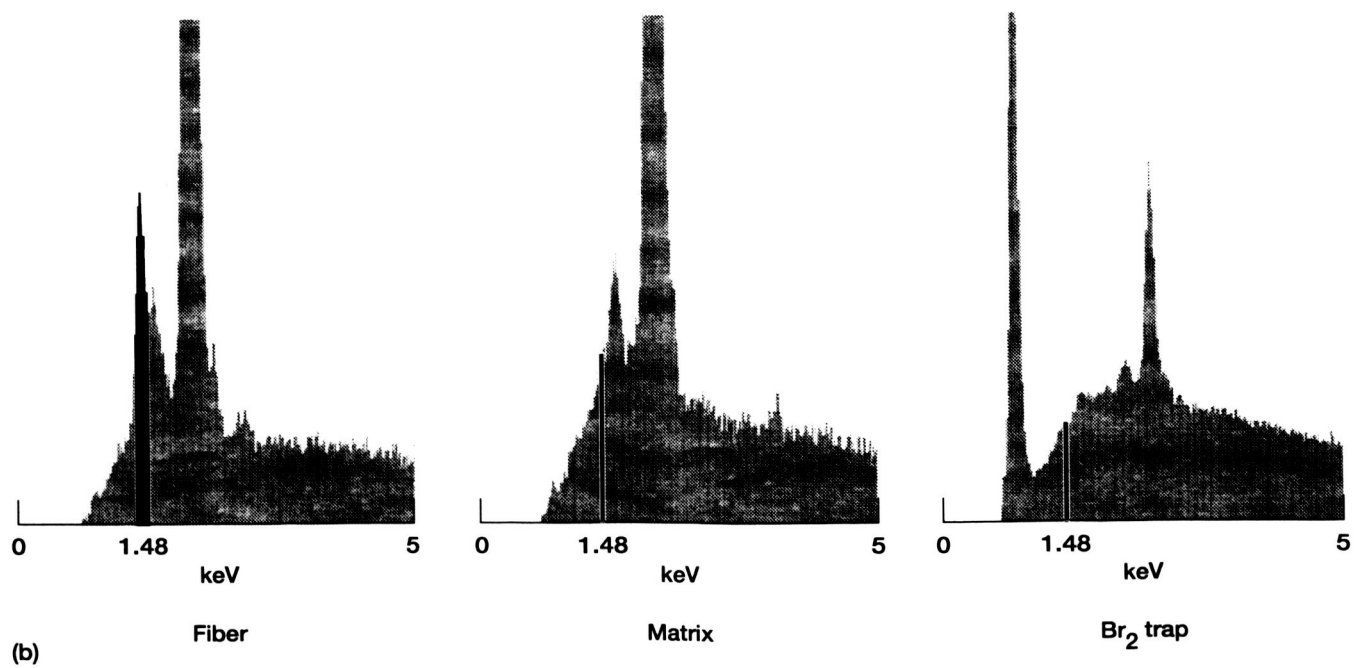
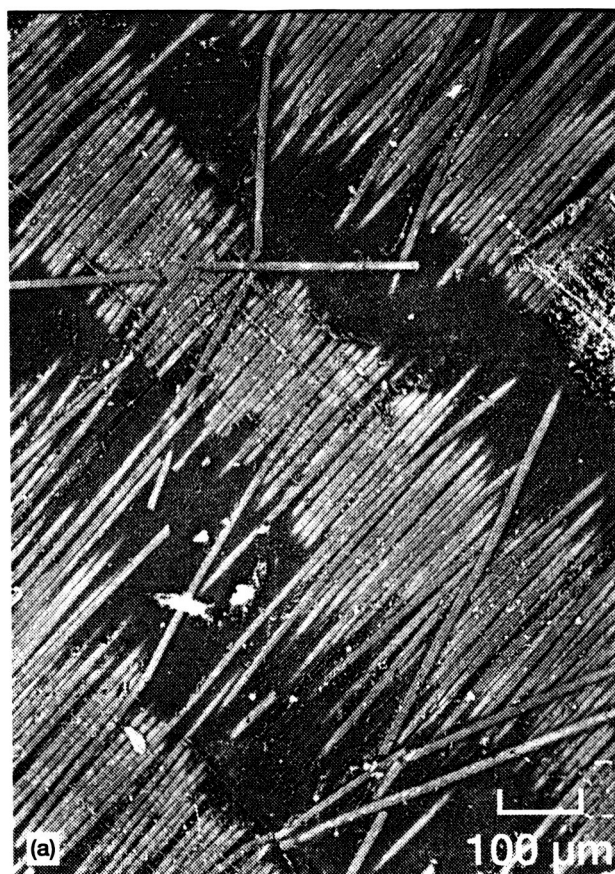


Figure 11.—Confinement of bromine to the fibers, (a) backscattering; and (b) energy dispersive x-ray spectroscopy.

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